INFRARED SPECTROSCOPY OF LARGE PARTICLES

THE INFRARED SPECTRA of relatively large particles can now be observed with a technique developed by the National Bureau of Standards.[1] Devised by Mary Harvey, B. G. Achhammer, and J. E. Stewart of the Bureau staff, the method involves suspending particles in a medium having approximately the same index of refraction as the particles themselves. From the resulting spectrum, changes in molecular structure can be determined. This procedure was worked out for use in a broad program now under way on the degradation mechanism in plastics.

Infrared absorption spectroscopy is being extensively utilized as a chemical tool for the analysis of large, complex molecules such as rubbers, plastics, and petroleum constituents. Infrared analysis techniques are also of value in correlating the molecular structure of these materials with their properties. Solid phase spectroscopy has required the particles analyzed to be smaller than the wavelength of incident radiant energy in order to minimize scatter. Therefore, plastic substances are ordinarily studied as thin films, cast from solution or pressed by applied heat. However, solvents often remain in the plastics and interfere with their spectra; the heat application is also unsuitable in some cases as it can cause thermal degradation.

Direct attempts to reduce the plastic particles to the established size requirements by grinding or mulling can also result in polymer breakdown. In the present method, the desired result—elimination of scatter—is achieved instead by matching the refractive indices of the particle and the suspension medium.

The plastic studied by this technique was polyvinyl chloride. Its index of refraction measured in white light by grain immersion methods is 1.548 at 27° C and apparently remains fairly constant with changing wavelength, a requirement for use of this method. However, all liquids of the proper index for use as suspension media for polyvinyl chloride exhibit infrared absorption bands, and a complete spectrum can be obtained only by using several suspension media. This situation led to further experiments using the recently proposed alkali halide pellet technique.[2] This method involves pressing an intimate mixture of alkali halide and the material under investigation into a pellet, which is then analyzed spectrographically. The method was modified to obtain the spectra of large particles by selecting an alkali halide with refractive index nearly matching that of polyvinyl chloride.

Potassium bromide, a solid that has a refractive index of 1.56 n_D^{20} and exhibits negligible absorption bands in the infrared, was used as the suspension medium. Finely ground and carefully dried potassium bromide was mixed with 0.028 g of polyvinyl chloride in a dry box and loaded into a die. Air was evacuated from the

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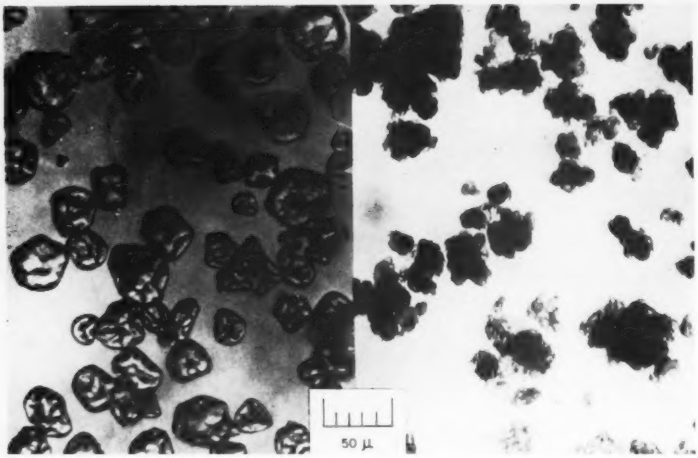


Attempts to reduce 44-μ particles of polyvinyl chloride (left) resulted in polymer breakdown, illustrated by the photograph at right. In both photographs 1 in. is equal to 80 μ.

die until a hard vacuum was obtained. The assembly was then subjected to a pressure of 95,000 psi for 7 min. In this way the potassium bromide-polyvinyl chloride mixture was formed into a clear pellet, ⅞ in. in diameter and about 0.05 in. thick.

The spectra of pellets produced in this way are free from scatter losses and contain no extraneous absorption bands. Spectra of polyvinyl chloride have been obtained using particles as large as 110 microns. Paper, nylon, and cotton fibers of large size, embedded in pellets of potassium bromide, have also yielded satisfactory spectra, demonstrating the utility of the method.

[1] *For further technical details, see* Index of refraction and particle size as factors in the infrared spectrophotometry of polyvinyl chloride, *by Mary Harvey, J. E. Stewart, and B. Achhammer, J. Research NBS* **56,** *225 (1956) RP2670.*

[2] The infrared and ultraviolet absorption spectra of cytosine and isocytosine in the solid state, *M. M. Stimson and M. J. O'Donnell, J. Am. Chem. Soc.* **74**, *1805 (1952)*; On the infrared spectroscopy of amino acids. I. A new preparation technique for infrared spectroscopy of amino acids and other polar compounds, *by U. Schiedt and H. Reinwein, Z. Naturforsch. [B]* **7,** *270 (1952).*

FALLOUT COMPUTER USED IN PACIFIC NUCLEAR TESTS

IN THE 1956 TESTS of nuclear devices in the Pacific Proving Grounds, the special-purpose electronic computer developed at the Bureau was used in predicting fallout. The original experimental model and, later, a duplicate were used in the operation. Although used primarily as a feasibility demonstration, the machines were of value in briefing military and scientific commanders at frequent intervals during the day prior to a scheduled detonation. The high speed of the computers made it possible to estimate the fallout pattern that would occur with the forecast winds and with various possible deviations from the forecast.

Safety is a prime consideration at nuclear bomb testing grounds. Precautions to prevent radioactive material from falling on inhabited areas require extensive information about the bomb and the weather. With this information, elaborate calculations can predict the fallout pattern. The two machines, specifically designed to perform these calculations, were used as safety aids by the fallout prediction group, which included J. H. Wright of NBS, and K. M. Nagler and K. Telegadas of the U. S. Weather Bureau. The development of the machines was sponsored by the Weather Bureau as part of its continuing research for the Atomic Energy Commission.

One of the machines was installed on a Naval command vessel, and the other at the island headquarters of the task force. Another type of machine, developed concurrently at the Sandia Corporation, was also successfully employed for the first time. A third type of machine, quite different from the others, is in an advanced stage of development at the Los Alamos Development Laboratory. The particular features of the NBS machine are its extremely high speed, virtually instantaneous display, and its adaptability to considerable variation in the formulation of the problem. However, as the operational accuracy of this type of machine had not yet been proven, ultimate reliance was placed in hand calculations performed by several scientists expert in the field of fallout prediction.

The NBS machine uses high-speed analog techniques in conjunction with a large cathode-ray display tube. On the face of the tube appears a map of the geographical distribution of radioactivity due to particles falling from an atomic bomb cloud. Input data consist of the size and shape of the cloud, radioactive distribution in the cloud, and the wind speeds and directions at 20 different altitudes. In its initial form, the machine appears to be useful up to a radius of about 200 miles from the burst point.

For further technical information about the radioactive fallout computer, see Radioactive fallout computer, *NBS Tech. News Bul.* **40,** *56 (April 1956)*; An analog computer for radioactive fallout prediction, *H. K. Skramstad and J. H. Wright, Proceedings of the National Simulation Conference, Dallas, Texas, January 21, 1956.*

X-RAY STUDIES OF PHOTOGRAPHIC FILM

EXPERIMENTS recently performed provide data on the photographic response of commercial X-ray films at unusually high rates of exposure.[1] Films of this kind are now used extensively for measuring X- and gamma-ray exposures. To interpret film densities in terms of exposure, however, a knowledge of film characteristics as determined under controlled laboratory conditions is required. By adding to this knowledge, the present study should help increase the reliability of such interpretations. The work was supported by the U. S. Atomic Energy Commission and the U. S. Army Signal Corps, and the experiments were conducted by Margarete Ehrlich and W. L. McLaughlin of the Bureau's radiation physics laboratory.

Previous studies had used exposure rates up to about 60 roentgens per second (r/sec) and a ratio of lowest to highest exposure rates of 1 to 10,000. The exposure rates used in the Bureau's experiments extended to about 1,100 r/sec, with a ratio of lowest to highest rates of 1 to 30,000. The results confirm the validity of the reciprocity law for exposures on the ascending branches of the characteristic curves of two widely used commercial X-ray films, over the exposure-rate range covered in the experiment. The results also establish the failure of the reciprocity law in the reversal region of the characteristic curve of one of the film types over the same exposure rate range.

In measuring the characteristics of X-ray films, the experiments dealt with reciprocity characteristics of film types widely used as detectors of X- and gamma radiation. The results have a bearing on the theory of basic photographic processes. The source of radiation is a 50-kv X-ray tube (vertical tube, right center) which sends an X-ray beam through a rotary shutter device to the film. Scientist is putting film sample in place near shutter opening. Rectangular box (partly visible, left center) is a free-air ionization chamber which, in conjunction with apparatus on table (front), measures the radiation intensity.

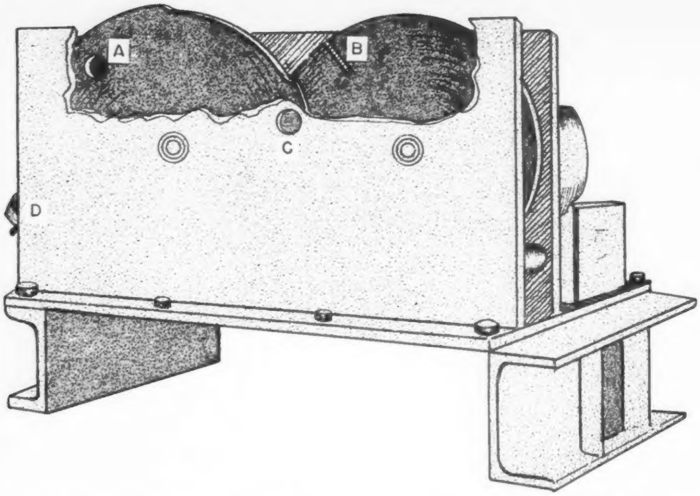
Film Characteristics

When a photographic emulsion is exposed to a source of radiation, the resulting photographic density of the developed negative depends on both the source intensity or rate of exposure, I, and the exposure time, t, other factors being equal. The reciprocity law states that the density of the negative is determined by the "total exposure" which is the product $(I \cdot t)$ of source intensity and exposure time. In many cases, however, as for example in the case of visible light, "reciprocity-law" failure occurs; that is, although the same total exposure is given, the density of the negative is not the same, but depends on the rate of exposure. As a rule, maximum photographic efficiency occurs at one particular exposure rate for a particular film type; whereas with other rates, longer exposure times are needed to produce the same photographic density.

The presence and degree of reciprocity-law failure can be deduced from a comparison of characteristic curves based on different rates of exposure. Such characteristic curves can be obtained by exposing the film at a constant exposure rate for various periods of time and measuring the photographic densities produced. The curves are usually plotted with density as ordinate and log $(I \cdot t)$ as abscissa. The reciprocity law fails to the extent that the characteristic curves based on different exposure rates indicate different densities for the same total exposure.

In the case of visible-light sources, deviations from the reciprocity law are the general rule. For X-rays, however, it has been generally suspected, and for certain rates of exposure experimentally established, that the reciprocity law holds very closely. An exception was expected for cases in which the X-rays produced luminescence in the emulsion. Because luminescent rays are in the wavelength range for which film shows reciprocity-law failure, such failure would be present if any considerable part of the X-radiation were converted to luminescent rays. An incidental result of the present investigation is that, for the exposure rates used, luminescence does not noticeably compete with the ordinary process of image formation.

For some time it has also been known that intermittent and continuous exposures may produce different densities, even when the total exposures are the same. In the experiments it was sometimes necessary to use an intermittent source (see below). There is a close connection, however, between the intermittency effect and reciprocity-law failure, the two effects being either both present or both absent.



Cutaway drawing of rotary shutter used in experiments on the validity of the photographic reciprocity law for X-rays. Steel disks containing hole (A) and slit (B) are rotated by synchronous motors at 100 and 3,600 rpm, respectively. When slit (B) passes hole (A) and both are in line with opening (C), X-rays enter at (C) and pass through to film on other side of shutter. This produces a 10-microsecond flash every 0.6 second. Shallow notch on left-hand disk actuates microswitch (D) so that number of flashes may be counted and total exposure controlled.

Another effect pertinent to the present experiments is "reversal," defined as a *decrease* in photographic density with increase in the total exposure beyond a certain level. Thus, if a photograph were taken with exposure levels in the reversal branch of the curve, the "negative" would actually be a positive.

The first part of the study dealt with the ascending branches of the characteristic curves, i. e., with the portions corresponding to total exposures less than the exposure required for maximum density. The other main part of the study was concerned principally with the reversal branches of the curves.

Ascending Branch

Two widely used X-ray films were investigated in the first part of the study. The source of radiation was a commercial 50-kv X-ray tube with a tungsten target and a 1-mm beryllium window. This apparatus produced the characteristic spectral lines of tungsten in the vicinity of 8 kev superimposed on a continuous spectrum with a peak at about 18 kev. Such a machine produces about 1 million roentgens per minute in the immediate vicinity of the X-ray tube window.

In order to achieve small total exposures with such a relatively powerful source, a rotary shutter arrangement was used in some cases. It consists of two partly overlapping steel disks of about 7 in. in diameter which are driven by synchronous motors at 100 and 3,600 rpm, respectively. One disk has a small circular opening near its periphery, and the other has a peripheral radial slit. The synchronization of the disk rotations was such that once every 0.6 sec both the circular opening and the slit were in line with the X-ray beam, so that flashes of about 10-μsec duration were produced. Special timing circuits were used to control the total exposures administered. When the rotary shutter was not employed its geometry was simulated by a diaphragming system.

Samples of both film types were exposed for varying periods of time at four different rates of exposure, obtained by adjusting the current in the X-ray tube. As measured by a free-air ionization chamber, the exposure rates were approximately 0.033, 1.3, 62, and 1,100 r/sec. All the exposures at 0.033 and 1.3 r/sec and all the exposures above 100 r at 62 r/sec were administered continuously. The other exposures were made with the help of the rotary shutter.

To test the effect of different photographic development processes, the experiments were repeated using conventional X-ray, internal, and surface developers. It is known that certain film emulsions will show reversal when processed in surface developer, whereas generally no such effect is found with the other more intensive types of developer. The present results conform to this general experience—reversal was found only with surface development and then only in 1 of the 2 film types studied.

After developing the films and measuring their photographic densities, the necessary data were available for plotting the characteristic curves. In all cases, the ascending branches were identical within the experimental error of ± 15 percent. Within the limits of experimental accuracy and for the range of exposure rates used, therefore, the reciprocity law was shown to be valid on the ascending branches of the characteristic curves of two typical X-ray films.

A detailed study of the curves, especially at the low-exposure end, indicated that even the ± 15 percent spread in the data cannot be due in any large measure to reciprocity-law failure. Also, when the films were exposed to gamma rays from a cobalt–60 source, a characteristic curve was obtained whose shape closely matched that produced by the 50-kv source. This suggests that the same results may hold for radiation energies much higher than those used for this study.

Reversal Branch

In the second part of the study, attention was confined to the film type that exhibited reversal effect, and only surface development was used. The experimental

setup and the four rates of exposure employed were the same, and all exposures in the reversal region were administered continuously except those at 1,100 r/sec, which were obtained both with continuous and with intermittent radiation.

For purposes of comparison, films of the same type were also exposed to visible light from a 500-watt floodlamp. The light exposures were administered continuously at several intensities (rates of exposure) in the range roughly from 1.7 to 1,700 meter candles. The different intensities were obtained with a calibrated neutral-density filter.

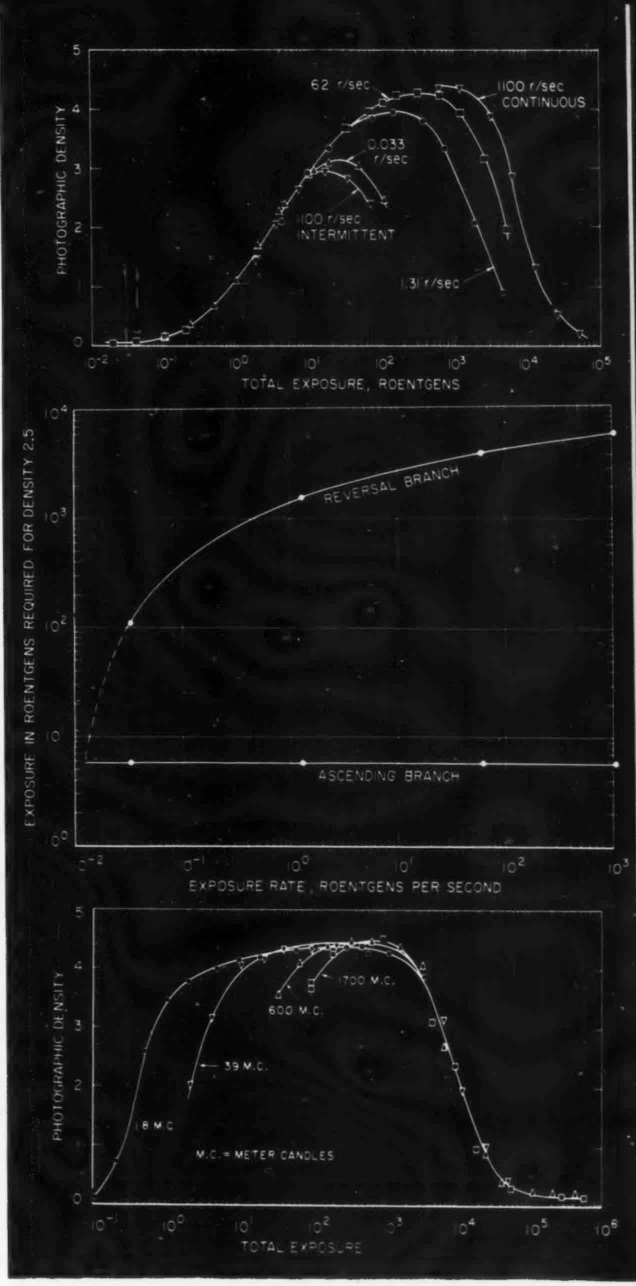
The characteristic curves obtained with X-radiation in the second phase of the study show that, for total exposures less than that required for reversal, all the curves coincide within the accuracy of the experiment. In agreement with the first part of the study, this again confirms the validity of the reciprocity law in the ascending branches for X-radiation over the range of exposure rates used.

The main result of the second set of experiments, however, is the experimental evidence for the presence of reciprocity-law failure and intermittency effect in the reversal branches of the X-ray curves. The first is shown not only by a shift of the reversal branches towards higher exposures as the exposure rate is increased, but by a marked change in the maximum density obtainable before the onset of reversal. The intermittency effect is revealed by the clear-cut difference between the reversal branch obtained at 1,100 r/sec with intermittent exposure and with continuous exposure. The position of the curve for intermittent exposure corresponded closely to what would have been obtained if the intermittent radiation had been replaced by continuous radiation at the same time-average rate.

In the case of visible light, failure of the reciprocity law for the ascending branches was confirmed by their shift toward higher total exposures as the rate of exposure was increased. On the other hand, the reversal branches were coincident under the present experimental conditions. Thus, in the present set of experiments, the reversal behavior for visible light was different from that for X-rays. However, earlier investigators had found that for certain exposure rates and processing conditions and for certain film types the reversal behavior with visible light may be of the type found here for X-rays. At present, no firm generalization appears possible on the connection between the reversal effect for X-rays and for visible light.

A satisfactory interpretation of the basic mechanisms involved must await further experimentation. The results of investigations at the Bureau and elsewhere suggest that further clarification of these problems could be expected from experiments both with visible light and with X-rays or high-energy electrons using exposure-rate intervals wider than those hitherto employed. Plans for further work in this direction are under consideration.

[1] *For further technical details, see* The reciprocity law for X-rays, Part I: Validity for high-intensity exposures in the negative region; Part II: Failure in the reversal region, *by M. Ehrlich and W. L. McLaughlin, J. Opt. Soc. Am.* **46**, *797, and 801 (Oct. 1956)*.



Top: **Characteristic curves of typical X-ray film obtained at various exposure rates. Rate of 1,100 r/sec was administered both continuously and intermittently (using rotary shutter); other exposures were continuous. Ascending branches of curves coincide, confirming validity of reciprocity law. Differences in maximum density and separation of curves after reaching maximum show reciprocity-law failure for the reversal branch. Intermittency effect is shown by difference between the two curves for 1,100 r/sec. *Bottom:* Response of same X-ray film to visible light. Intensities (in meter candles) used in obtaining each of the curves are approximate. Coincidence of reversal branches, indicating validity of reciprocity law for that part of characteristic curve, may be due to extraneous factors. *Center:* Total exposure to X-rays needed to produce the same photographic density in a typical X-ray film at various exposure rates. If reciprocity law were valid, a horizontal line would be obtained. Graph shows the law holds for ascending branch but fails for reversal branch.**

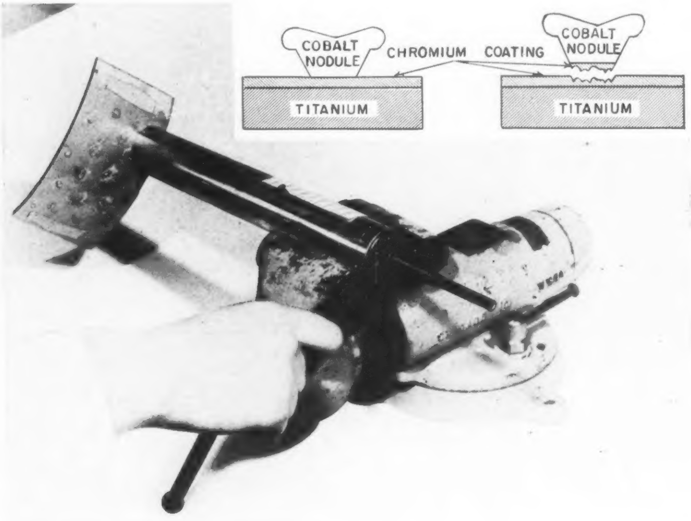
Protective Coatings for Titanium

THE BUREAU has successfully electrodeposited hard adherent protective coatings on titanium. The procedure involves forming a titanium fluoride film on the metal surface, electroplating with chromium, and heat-treating the plated specimen at 800° C.[1] Developed for the Springfield Armory by C. L. Stanley and A. Brenner of the Bureau staff, the process is expected to extend considerably the utility of titanium metal, particularly for high-temperature applications.

Because of titanium's high strength-to-weight ratio, it ranks with steel and aluminum as a structural material. However, titanium has disadvantages for some applications: It tends to gall or seize when in loaded contact with itself or other metals, and it oxidizes at elevated temperatures. These disadvantages could be minimized if titanium were coated with hard, oxidation-resistant metals such as chromium or nickel, but previous attempts to produce such coatings have not been entirely successful. Although the Bureau had obtained good adhesion of aluminum to titanium in an earlier investigation using a nonaqueous plating bath, the method was not suitable for job-shop applications and the results were not sufficiently consistent.

The main problem in depositing metals on titanium is lack of adhesion—some deposits have actually exfoliated during the plating operation and others have been easily pulled off with the fingers. Poor adhesion has been blamed on an oxide film on the base metal, but attempts to remove the suspected film with acid etches or anodic films were not successful. For this reason, the present investigation also studied the influence of oxide films and methods to remove them.

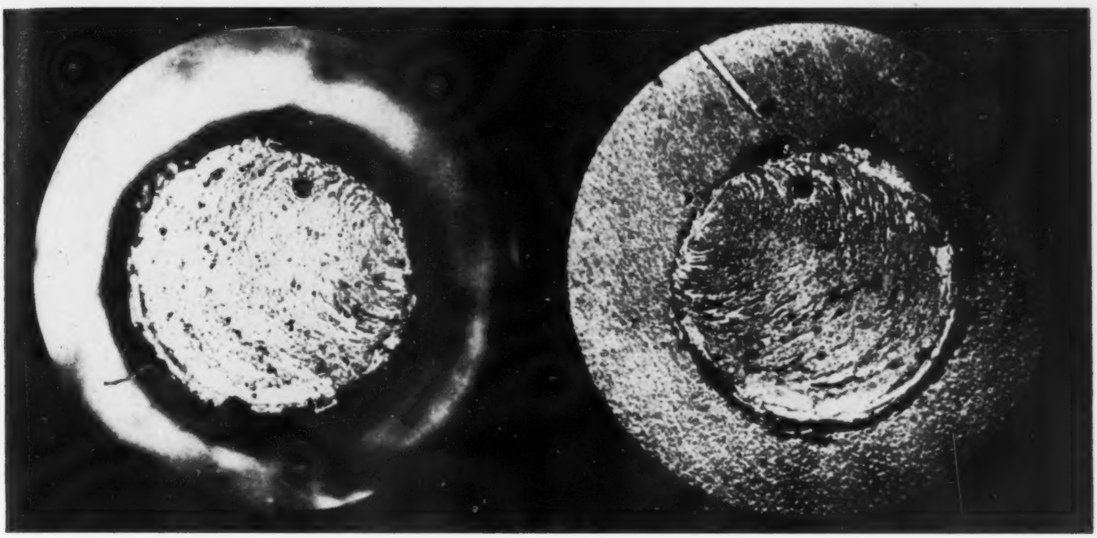
In a preliminary investigation, the Bureau designed an experiment intended to remove any oxide film and to plate the titanium specimen before it could reoxidize. In this procedure, a small piece of titanium was enclosed in an evacuated tube containing silicon carbide and ceramic balls. The tube was tumbled for several hours to abrade the metal surface and then placed in a chromium plating bath, where it was crushed so as to expose the metal specimen to the bath before the atmosphere could touch it. In a control experiment, a titanium specimen was similarly abraded in an open tube. The adhesion of the first specimen was distinctly better than that of the control, supporting the hypothesis that a film, probably an oxide, exists on the surface of titanium and impairs adhesion of metal deposits.



Apparatus for removing cobalt nodule in test for adhesion of electrodeposited metal coatings. The nodule is gripped in a chuck attached to a spring balance and detached by exerting force through the spring. The adhesive force per unit area is computed from a reading of the balance. Diagram illustrates cross section of nodule on chromium-plated titanium specimen before and after fracture.

The Bureau investigated a number of etching and plating procedures and obtained the best chromium plates by pretreating titanium to form a coating of titanium fluoride, before plating the specimen. This procedure appears to prevent the formation of an oxide and, when the specimen is placed in the plating bath, the titanium fluoride dissolves permitting the chromium to bond directly to the basis metal.

Titanium specimens are thoroughly degreased and cleaned before this treatment. Next they are dried and suspended in a solution of hydrofluoric and acetic acids. After 10 or 15 minutes a 60-cycle alternating current is passed through the specimen for another 10 minutes. The specimens are then rinsed and transferred to a conventional chromium plating bath, where they are plated at a temperature of 85° C and a current density of 120 amp/dm^2.



Chemical analysis indicated that the film produced by the preliminary acid treatment contains a low valence titanium compound. A sample of the dried film contained 37 percent of titanium and 54 percent of fluorine, and examination by X-ray diffraction showed no lines characteristic of titanium tetrafluoride.

Tensile strength tests indicate that adherence of the chromium coating to titanium is greatly improved by heat-treating the plated specimen for two minutes at 800° C in an inert atmosphere. Coating adhesion was determined by the nodule method.[2] Essentially this procedure consists in electrodepositing a cobalt nodule, 1/16 in. in diameter, on the coating and then determining the force necessary to detach the nodule together with the coating from the basis metal. The bond strength of the heat-treated specimens ranged from 4,000 to 18,000 lb/in.^{2} Although the highest value obtained is somewhat below the tensile strength of heat-treated chromium, fracture occurred in all cases in the plating and not between the two metals.

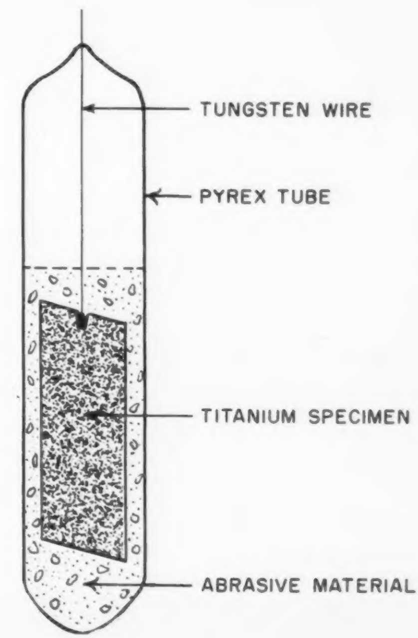
Coatings on titanium consisting of 0.02 mm of chromium plus 0.15 mm of nickel were found to be moderately adherent without heat treatment, but not comparable to that of chromium on steel. If such specimens 0.5 in. wide are broken by repeated bending, the chromium-nickel coatings can be stripped from the titanium with a force of about 10 pounds.

Procedures for depositing nickel and copper on titanium have been formulated that are similar to the process for depositing chromium. However, the nickel and copper deposits have not adhered as well as chromium and have frequently blistered.

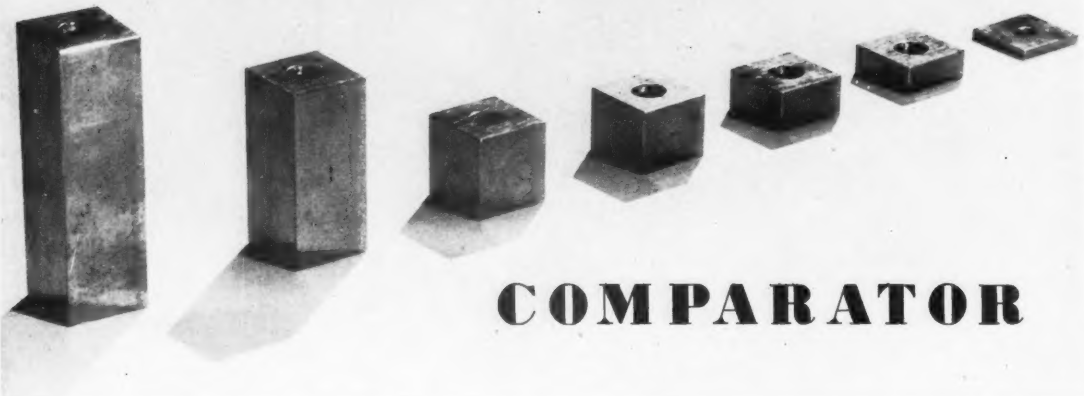
[1] *For further technical details, see* The adhesion of electroplated coatings to titanium, *by Connie L. Stanley and Abner Brenner, Tech. Proc. Am. Electroplaters' Soc., 1956.*

[2] *See* The nodule method of measuring the adhesion of electrodeposited coatings, *by Abner Brenner and Virginia D. Hills, Proc. Am. Electroplaters Soc.* **37**, *51 (1950); also,* Nodule method measures adhesion of electrodeposits, *NBS Tech. News Bul.* **34**, *80 (June 1951).*

Photomicrographs of fractured metal surfaces after nodule test for adhesion of heat-treated chromium on titanium. The fracture has occurred in the chromium, leaving some still bonded to the titanium. *Left:* Nodule surface. *Right:* Specimen surface. *Below:* Diagram of glass tube assembly used to investigate the effect of oxide film on plating titanium. The specimen is suspended from a tungsten wire in the evacuated tube. The tube is agitated so that abrasives in the tube remove any film from the specimen. Then the tube is placed in a regular chromium plating bath and crushed with pincers so that plating begins before the titanium can reoxidize.



GAGE-BLOCK COMPARATOR



An interferometric comparator that makes routine comparisons of length to the nearest ten-millionth of an inch has been developed. Designed by T. R. Young and J. B. Saunders of the Bureau's engineering metrology laboratory, the extremely precise instrument [1] will be used to check lengths of industry's master gage blocks which control the tolerances of mass-produced machine parts. An instrument of this accuracy has been greatly needed because of the extremely small dimensional tolerances now required for parts used in the guided-missile, jet-aircraft, machine-tool, and other industries.

At present the Bureau calibrates master gage blocks to an accuracy of 1 part in a million, that is, to the nearest millionth of an inch for inch-long blocks. However, the use of continually smaller tolerances in industry has caused machine-tool manufacturers to request that the Bureau develop procedures for calibrating their master gage blocks on a routine basis to 1 part in 10 million. Because of the extreme importance of this work, the Congress has provided a substantial increase in the Bureau's appropriation, specifically requesting increased effort on the project. Ten major industrial firms [2] have also made available a grant of $19,500 per year for 3 years so that the necessary research can be carried out as soon as possible.

Development of the precise interferometric comparator represents an important step toward the goal of this program. A major problem yet remaining is the development of means for absolute calibration, to the nearest ten-millionth of an inch, of the primary standard gage blocks with which the Bureau compares industry's master gage blocks. The Bureau is also making a study of gage-block materials in connection with this program.

Like other interferometers, the high-precision comparator determines the relative positions of reflecting surfaces by means of interference fringes formed by light reflected from these surfaces. However, in this instrument readings are taken directly from a micrometer screw. Thus, greater accuracy is obtained by eliminating visual estimation of fringe displacement.

As the interference fringes can be produced with white light instead of the usual monochromatic sources, the measurement does not depend on the wavelength of light used. This, in turn, greatly simplifies the measuring procedure so that the training required of the operator is minimized. Other advantages of the design are high sensitivity and less instability due to vibration.

The two gage blocks being compared are wrung to the horizontal surface of a flat plate so that they stand on end, side by side, above the plate. A double prism transmits two beams of collimated white light down onto the top surfaces of the gage blocks and the flat plate. After reflection from these surfaces the two beams are combined at the dividing surface of the double prism, and two separate patterns of interference fringes are formed in the eyepiece of a telescope. One fringe pattern is within the outline of the superposed images of the gage blocks; the other is formed by the images of the flat plate. If the gage blocks are of equal length, the two fringe patterns will coincide, but if there is a difference in length, the fringe patterns will be proportionately displaced with respect to each other. The observer then uses a micrometer screw to move portions of an optical wedge system until the two fringe patterns successively coincide with the cross hair in the microscope eyepiece. The distance from one point of coincidence to the other is read from the micrometer and converted into the corresponding difference in gage-block length by means of a previously determined calibration constant.

In comparing the fringe patterns for coincidence, no prior knowledge of the approximate difference in gage-block length is required to avoid misinterpretation of interference order. The white light pattern provides a characteristic color for the reference fringe in each pattern. Thus the observer has only to rotate the micrometer screw until both reference fringes coincide with the cross hair in the eyepiece.

The optical wedge system consists of four wedges, two in each of the interfering beams. The wedges that are paired together are matched in both wedge angle and index of refraction. One wedge from each pair is attached to a movable stage driven by the micrometer screw. Thus, the wedge system in effect introduces a plane parallel plate of variable thickness in each beam. Rotating the micrometer screw through the proper distance then causes a differential change in the optical paths of the two beams, which brings the interference patterns into coincidence with the cross hair.

The wedge angles can be chosen so as to provide a convenient calibration constant between micrometer screw rotation and the change in optical path. In the instrument constructed at the Bureau, the distance indicated on the micrometer is the difference in gage-block length multiplied by 5,000.

To keep this calibration constant invariant (or nearly invariant) for the range of visible wavelengths included in the white light (tungsten illumination) used, the wedge pairs are made of glasses having different indices of refraction. Then, to compensate for the glass of the wedges in the opposing beam, a plane parallel plate is inserted in each beam. Each plate has an index of refraction equal to that of the wedge system in the opposing beam when that wedge combination has been adjusted to compensate for a zero path difference in air. Use of the parallel plates is necessary to retain the white light interference condition.

Experiments have shown that the calibration constant is sufficiently invariant with wavelength to allow the full range of the instrument to be used to compare gage blocks differing in length by as much as 2 ten-thousandths of an inch.

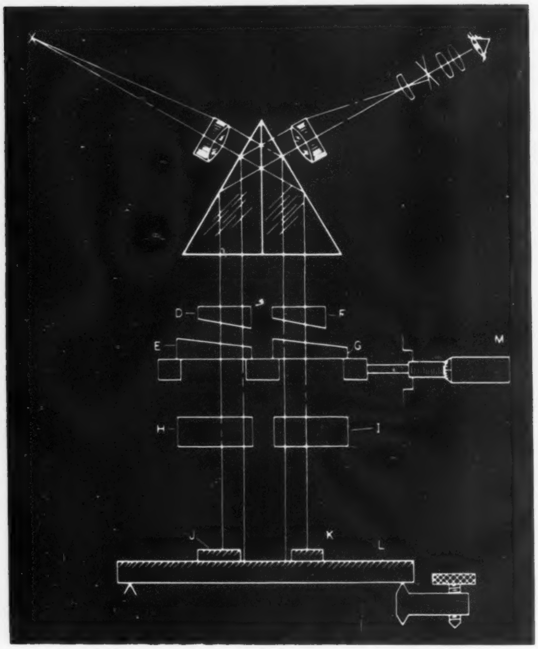
To determine the precision attainable in practice, the interferometer was used to compare the lengths of 10 gage blocks differing in nominal length by increments of one millionth of an inch. In the comparisons a statistical design developed in the statistical engineering laboratory was employed. This schedule of observations [3] provides values for comparisons of the 10 gage blocks in all possible pairings even though only a limited number of pairings are actually measured directly. The length difference for each of the pairs is obtained as a simple least square solution that is free of bias due to experimental, instrumental, or observational characteristics. The over-all results are thus well balanced and favor no particular comparison.

The results of the gage-block comparisons indicated that the least square solution for the lengths of the gage blocks had a probable error of 3 one-hundred-millionths of an inch. The largest spread between measured comparisons and the least square solution was 1.1 ten-millionths of an inch with a probable error for each measured comparison of 8 one-hundred-millionths of an inch. Thus, the evidence shows that the comparator can be used to make individual comparisons of gage blocks, such as are ordinarily required in comparing a gage block with a standard, to a precision of 1 ten-millionth of an inch.

The results of this experiment will also serve as a guide in designing a second model of the interferometer to measure the absolute length of the Bureau's primary standard gage blocks. The experiment demonstrates that the precision of alinement of the reference fringe with the cross hair is such as to allow for a probable error of 8 one-hundred-millionths of an inch. On this basis, computations show that in order to hold the overall probable error to 1 ten-millionth of an inch, the probable error resulting from the additional variables involved in measuring absolute length should not exceed 6 one-hundred-millionths of an inch.

Variables that must be accurately controlled in the absolute measurement include pressure, temperature, and humidity of the air; temperature and thermal ex-

This high-precision interferometer compares gage-block lengths with an accuracy of 1 part in 10 million. The gage blocks rest on the circular flat plate below. White light from the source at top is reflected from the gage blocks and flat plate to produce two different interference patterns in the telescope eyepiece (circle). The inner rectangular pattern is within the superposed images of the two gage blocks; the outer circular pattern results from the images of the flat plate. The difference in the lengths of the gage blocks is determined in terms of the rotation of the micrometer screw (left of center) required to bring the two interference patterns successively into coincidence with the cross hair.



Schematic diagram of the interferometric comparator developed for precise comparison of gage blocks. White light from a point source (upper left) is first collimated by an achromatic lens. It then enters the double prism, where it is separated into two parallel beams. The two beams are reflected normally from the top surfaces of the gage blocks J and K, and also from the flat plate L, to which the gage blocks are wrung. After reflection the two beams are combined at the dividing surface of the double prism, and two separate patterns of interference fringes are formed in the telescope eyepiece (upper right). The difference in gage-block lengths is measured by the horizontal distance through which optical wedges E and G must be moved to bring the two interference patterns successively into coincidence with the cross hair. This distance is obtained from the differential reading of the micrometer screw M, which moves the horizontal stage supporting wedges E and G.

pansion coefficient of the gage-block material; and the phase change occurring at reflection from the surfaces of the gage block and the flat plate. This phase change is a function of the material and the surface finish.

For routine comparison of gage blocks, the comparator design makes the variables mentioned above much less significant. For example, the optical paths and the gage blocks being measured are in close proximity, which minimizes temperature effects. In addition, the gage blocks may be conveniently interchanged between the two light beams. This procedure cancels out the effects of constant temperature gradients and residual effects due to the optics of the interferometer. Also, the fact that measurements are made at two points closely spaced in the field of the interferometer imposes less stringent requirements for constancy of surface on the optical parts and the flat plate.

[1] *For further technical details, see* Achromatic interferometer for gage block comparison, *by T. R. Young, Proceedings of a Symposium on Gage Blocks Held at the National Bureau of Standards on August 11 and 12, 1955, NBS Circular (in press).*

[2] *See* Gage block research conference, *NBS Tech. News Bul.* **40**, *141 (Oct. 1956). The manufacturing firms supporting the Bureau gage block research program are E. I. du Pont de Nemours & Co., Greenfield Tap & Die Corporation, Hughes Aircraft Co., The Taft-Peirce Manufacturing Co., The Timken Roller Bearing Co., The Van Keuren Co., The Sheffield Corporation, The DoAll Co., Dearborn Gage Co., and Pratt & Whitney Co., Inc.*

[3] New experimental designs for paired observations, *by W. J. Youden and W. S. Connor, J. Research NBS* **53**, *191 (1954) RP2532. See also* Comparisons made in pairs, *NBS Tech. News Bul.* **39**, *20 (Feb. 1955).*

Standard Scale of Sugar Color

ACCURATE determination of color is of great practical importance in the sugar industry. Since early in the 19th century, estimation of color has been used to evaluate liquid sugar products, to detect the changes that occur during processing, and to evaluate adsorbents for removal of sugar color. However, the successful use of color measurements in the sugar industry has been continually handicapped by a lack of standards of sugar color and a suitable color unit. During the last 50 years many reference materials have been proposed for this purpose but none has proved satisfactory in all respects.

Recently a fundamental unit of sugar color [1] was developed by members of the Bone Char Research Project, Inc., a research fellowship maintained at the Bureau by manufacturers of cane sugar and of bone char for sugar refining. This unit of sugar color, together with its associated color scale, has been used successfully to characterize a number of typical sugar solutions.

The NBS scale of sugar color is obtained by using an empirical formula to evaluate the perceptible difference between the color of a commercial sugar and that of a highly purified colorless sucrose solution. Such a scale permits the use of a single number to represent visual appearance. It thus furnishes the sugar industry with the simplest possible characterization of color, and it places the measurement on a relative basis like that used in industry.

In practice the color of a sugar solution in NBS units is obtained from a simple chart without calculation, once the transmittancy of the solution at two wavelengths, 420 and 560 millimicrons, is known. This is possible because of the inherent simplicity of sugar

colors as shown by spectrophotometric curves of transmittancy versus wavelength for sugar solutions. In these curves, the transmittancy always increases with wavelength, and a simple algebraic relationship is found to exist between transmittancy and wavelength. This relationship makes it possible to construct the complete spectrophotometric curve for a given solution from transmittancy observations at only two wavelengths.

The color chart consists of a series of plotted curves, each curve representing a given value of sugar color in NBS units. The two axes on which the curves are plotted are graduated in terms of the attenuancy [2] (reciprocal of transmittancy) of a sugar solution at 420 mμ and 560 mμ, respectively. Thus, any pair of values of transmittancy at the two wavelengths determines a point on the chart. If the point falls on one of the curves, its value in units of sugar color is obtained from the curve; otherwise its value is found by interpolation.

In plotting the curves on the color chart, the values of sugar color in terms of attenuancy were computed by means of the Adams formula [3] for color difference. For this purpose sugar color was defined as the difference in color between the measured solution and the standard colorless sucrose solution. The Adams formula is an empirical relationship which has been used quite successfully for calculating color differences between nearly equal colors of two reflecting surfaces. Although the calculated differences for sugar color are not small, experience indicates that this is not a substantive limitation provided differences greater than 40 NBS units are not involved. Larger magnitudes are avoided by specifying the depth of solution to be employed in the measurement.

The color chart has been checked in several ways and found to be in good agreement with visual observation. For example, a series of sugar solutions was prepared and tubes containing equal depths of the solutions were alined by independent observers in a sequence of decreasing color. It was found that this sequence was substantially identical with that determined from values of the color in NBS sugar units. Statistical analysis of the comparison indicates that the values in NBS units can be used with confidence by the sugar industry in grading the color of normal sugar solutions.

One additional useful property of the new scale of sugar color is additivity. This makes it possible to predict the color of a mixture from those of its components.

The NBS unit of sugar color is calculated from the transmitted light only. Caution must therefore be exercised in visual comparisons to take into account the light reflected from container walls. When this is done, the NBS unit fulfills the need for a primary color standard in the sugar industry. Secondary standards, such as suitable glasses, may also be found useful.

[1] *For further technical details, see* Color evaluation in the cane sugar industry, *by Victor R. Deitz, J. Research NBS* **57**, *159–170 (1956) RP2706.*

[2] *The term "attenuancy" has been introduced to replace "absorbancy" for those solutions that contain appreciable light-scattering material.*

[3] Color in business, science, and industry, *by D. B. Judd (John Wiley & Sons, Inc., New York, N. Y., 1952).*

Sugar solutions are placed in a spectrophotometer for transmittancy measurements at the National Bureau of Standards. Once the transmittancy of the solution is known at two wavelengths, a simple chart gives the color of the solution in NBS units without calculation. Standard commercial equipment is used throughout the measurement.

ECHO-FREE CHAMBER

A NEW anechoic chamber or "deadroom" of modern design has been added to the facilities of the Bureau's sound laboratory. Within the chamber, echoes in the audiofrequency range are minimized by an elaborate acoustic treatment of glass fiber wedges, which cover the walls, ceiling, and floor. The acoustic design was worked out by W. Koidan of the sound laboratory, the wedges were manufactured and installed by The Eckel Corp., of Cambridge, Mass., and the construction work was directed by the Bureau's Plant Division.

The deadroom will be used for experiments requiring an environment essentially free from echoes. This condition insures that practically all of the sound energy arriving at a microphone comes directly from the original sound source and that only a negligible amount reaches the microphone by reflections from the room surfaces. Reflections of sound from walls, ceiling, or floor would give rise to erroneous results, for example, in measuring the response characteristics of a sound source with a calibrated microphone. Anechoic conditions are also essential when comparing the free-field responses of different sizes or types of microphones, such as velocity-type versus pressure-type. If free-field conditions did not prevail, the relative sensitivities of the microphones would appear to depend on the environment in which the measurement is made, so that it would be almost impossible to perform the calibration work.

To achieve the echo-free condition, the surfaces of the room are lined with wedges of matted glass fibers, each wedge being 40 in. deep from its tapered end to its 8-by-24-in. base. Held in shape by an open wire netting, the wedges are fabricated in units of three each. The units are mounted so that the tapered edges of each unit point into the room at right angles to those of adjacent units. The units are attached to a wooden cellular framework which also provides a 6-in. airspace between the wall and the bases of the wedges. To attenuate electromagnetic disturbances from outside, such as radiation from nearby television stations, the interior faces of the walls are lined with a 0.003-in. layer of copper shielding.

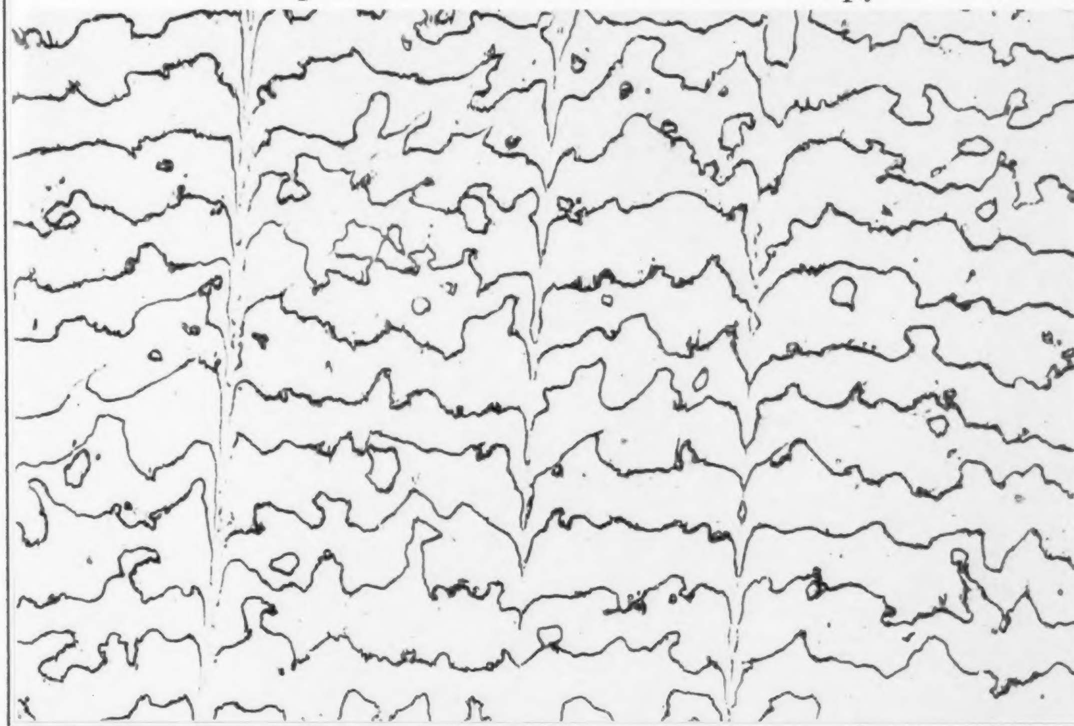
The working space in the room is approximately 21 by 16 by 10 ft. The door, 48 in. thick, has the same acoustic treatment as the walls and moves easily in and out along overhead rails. A horizontal, interlaced steel-cable net makes it possible to walk about in the room just over the floor wedges. About $\frac{3}{32}$ in. in diameter, the individual cables are attached through compression springs to I-beams that ring the lower part of the room.

Interior of newly built echo-free chamber or "deadroom." Wedges of matted glass fibers which line the walls, ceiling, and floor absorb about 99 percent of incident sound energy over most of the audiofrequency range. Horizontal net of thin steel cables permits personnel to walk about in room just above floor wedges. The sound source near left wall and the microphone, which scientist is attaching to a preamplifier, are being used in tests to determine the acoustic characteristics of the chamber. The room will be used for such purposes as the calibration of microphones and sound sources.

Tests are under way to determine the room's characteristics. These tests will provide useful data on this type of construction and will enable personnel using the room to take its characteristics into account when making acoustic measurements. After the tests are satisfactorily completed, the room will be used in the Bureau's regular research program in sound measurement, and will be available to a limited extent to Government and private agencies requiring secondary calibrations of laboratory sound equipment.

Some of the intended uses include the secondary free-field calibration of microphones; research in the scattering of sound; the calibration of sound-level meters; the testing of hearing aids; the measurement of noise produced by certain types of apparatus, such as ventilating fans; and an investigation of the characteristics of sound sources, with the aim of developing a stable sound source suitable for laboratory work.

Multiple Beam Interference Microscopy



This photograph was awarded First Prize in the General Photomicrographs, Non-Metallic Group of the 10th American Society for Testing Materials Photographic Exhibit and Competition, June 1956. It shows the interference pattern obtained when a sheet of crazed polymethyl methacrylate is used as the specimen surface for a multiple-beam interferometer. S. B. Newman and Irvin Wolock have adapted this procedure to determination of surface dislocations in plastic sheets. Both the plastic specimen and the optical flat used as a test surface were metalized. When one surface was placed one on top of the other, the irregularities in the crazed material caused corresponding irregularities in the interference pattern. Deviations from the horizontal indicate height differences in the polymethyl methacrylate surface. Thus, each vertical series of V-shaped configurations represents the site of a crack. The light source was the 546-μ line of a mercury lamp (linear magnification, $\times$80).

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Journal of Research of the National Bureau of Standards, Volume **57**, No. 5, November 1956 (RP2716 to RP2719 incl.), 60 cents. Annual subscription $4.00.

Technical News Bulletin, Volume **40**, No. 11, November 1956. 10 cents. Annual subscription $1.00.

Basic Radio Propagation Predictions for February 1957. Three months in advance. CRPL-D 147. Issued November 1956. 10 cents. Annual subscription $1.00.

Research Papers

Journal of Research, volume **57**, *No. 5, November 1956. Single copies of the Journal, 60 cents. Single copies of Research Papers appearing in the Journal are not available for sale. The Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C., will reprint 100 or more copies of a Research Paper. Request for the purchase price should be mailed promptly to that office.*

RP2716. Efficiency of 4π-crystal-scintillation counting: 1. Experimental technique and results. C. C. Smith, H. H. Seliger, and J. Steyn.

RP2717. Efficiency of 4π-crystal-scintillation counting: 2. Dead-time and coincidence corrections. W. B. Mann and H. H. Seliger.

RP2718. Entropy changes in rarefaction waves. Robert F. Dressler.

RP2719. Statistical investigation of the fatigue life of deep-groove ball bearings. J. Lieblein and M. Zelen.

Circulars

C539. Volume 6. Standard X-ray diffraction powder patterns. Howard E. Swanson, Nancy T. Gilfrich, and Marlene I. Cook. 40 cents.

C575. Bibliography on nitrogen 15. Margaret W. Chapman and Herbert P. Broida. 15 cents.

Patents

(The following U. S. Patents have been granted to NBS inventors. Assigned to the United States of America, as represented by the Secretary of the department noted in parentheses.)

No. 2,752,615. July 3, 1956. Marker buoy. Leland L. Parker. (Navy).

No. 2,753,449. July 3, 1956. Superheterodyne mixer with negative feedback for stabilizing conversion gain. Gail E. Boggs. (Commerce).

No. 2,754,180. July 10, 1956. Apparatus for growing single crystals and purifying substances. Avery T. Horton. (Commerce).

No. 2,761,968. September 4, 1956. Electronic analogue-to-digital converters. Milton L. Kuder. (Commerce).

No. 2,761,971. September 4, 1956. Crystal-controlled blocking oscillators. Moody C. Thompson, Jr. (Commerce).

No. 2,762,858. September 11, 1956. Punched-cell wax electrolyte batteries. Reuben E. Wood. (Commerce).

No. 2,763,001. September 11, 1956. Reflected-ray eliminators. Howard E. Bussey. (Commerce).

No. 2,764,734. September 25, 1956. Phase angle method of metal thickness indication. Wilfrid A. Yates. (Commerce).

(The following U. S. Patents have been granted to NBS inventors. Licensed to the United States of America, as represented by the Secretary of the department noted in parentheses.)

No. 2,754,711. July 17, 1956. Screw cutting attachment. Gustave Shapiro, Robert O. Stone, and Robert L. Henry (Navy).

No. 2,762,566. September 11, 1956. Code matching systems. Joshua Stern. (Commerce).

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